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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the directions of the slurry trash produced from production of a silicon wafer. This invention relates to the approach for using the solid-state fraction of the slurry produced in case it saw-lengthens or a silicon wafer is sliced from the silicon ingot as a raw material in industrial processes, such as production of the specific ceramics, more at a detail.

[0002]

[Description of the Prior Art] The volume in the world of the high purity silicon has reached 20,000 metric tons [per year] (MT/year) order now. This silicon is consumed by the semiconductor industry for mainly producing the photovoltaics industry and the integrated circuit for producing a solar battery.

[0003] An integrated circuit is the past dozens of years, and has become a very common resource very importantly. In advanced nations, substantially, everyone can all use the electronic equipment containing an integrated circuit, and/or own such electronic equipment. The indication which shows that this importance decreases in the future [of the range which can be predicted] does not have anything. With it, reversely, generally, an electronic instrument spreads more than former, and it is thought that it will become in a part indispensable to our life.

[0004] Although a solar panel still is not common in everyday life, the latest development of a solar panel has produced the panel with the conversion rate from the solar energy which deserves attention to electrical energy. Therefore, it is predicted as that from which a solar panel generally becomes the source of electrical energy used widely. This judgment has found out the support which increases increasingly from the interest about the climate change which human being brought about by dependence to combustion of the fossil fuel in the advanced nations for producing the energy which we use increasing.

[0005] Therefore, both integrated circuit and solar panel are considered to contribute to the violent rise of need to the silicon of the high grade in the near future. Moreover, both an integrated circuit and a solar battery are made from the ingot of the pure silicon sliced by the configuration of a thin disk or a wafer. Probably, things various from hundreds of microns to several mm have the thickness of these wafers. There are two main things in the technique used today in order to slice the silicon ingot of a high grade in the configuration of a wafer fundamentally. They are a wire saw and a diamond saw. However, importance and a market share with an increasing wire saw are obtained.

[0006] In the case of a wire saw, an ingot is sliced by persisting in them to the web of the wire made from steel which moves by the high speed by which coating was carried out in the layer of brass. Among a slice process, a wire is either the glycol which contains SiC as a polish particle, or mineral oil, and is always rinsed. This actuation brings about the slurry which used as the base the oil or the glycol which cuts with a SiC particle and contains slot residue (small particle of a high grade Si).

[0007] In the case of a diamond saw, a slurry consists of a pure silicon chip and water. If soaked in water, as for the silicon chip, the front face will oxidize. Like aluminum, only some atomic layers of the surface section will oxidize and the oxygen non-permeated layer which consists of SiO₂ will be formed.

Therefore, oxidation will stop automatically.

[0008] Since the thickness of a wafer and a solar battery is thin, the amount of end slot loss also becomes 40wt% among the overall rates of consumption of the high purity silicon. Typically, end slot residue exists in the amount of the range of 10 - 40wt% in a slurry. Therefore, the current annual amount of slurry generation also becomes 10 - 15000MT / year, and it is expected that this numeric value is taken for passing through a year from now on, and increases violently.

[0009] Slurry trash is deposited on current and a reclaimed ground, is burned with the incinerator, therefore constitutes the big trash problem. The approach of depositing a slurry on a reclaimed ground is a solution dangerous in environment from there being a danger that oil and a glycol will exude in soil. For this reason, by such reclamation, the need of devising the environment-edfety to which expense increases is searched for. Although the approach of incinerating solves the problem about the soil pollution by oil or the glycol, the silicon end slot residue in a slurry is changed into polluted SiO (accompanied by the heavy element in many cases)², and must deposit it with SiC this polluted SiO₂ of whose is also the third component of a slurry.

[0010] This solution over a slurry trash problem is obtained from both economical viewpoint and environment-viewpoint, and is a regrettable solution. If these end slot residue and SiC particles are the residua from what belongs to an available raw material with the highest purity commercially, therefore it takes into consideration especially that they constitute the valuable ingredient which was suitable for applying as a raw material in many industrial applications enough, it is still more so.

[0011]

[Problem(s) to be Solved by the Invention] The first object of this invention is abolishing the need of disposing of the trash by using as a raw material for applying slurry trash in other industries.

[0012] The second object of this invention is offering the approach for carrying out the reuse of the solid-state fraction of this slurry-like sludge produced in saw length of silicon as a raw material in production of the ceramics.

[0013] The technique indicated by the term of an attached claim and/or the following explanation can attain these objects of this invention.

[0014] Moreover, the above-mentioned object of this invention can be attained by utilizing the data that the solid-state part of the slurry produced from the wire saw which consists of mixture of Si particle of a very detailed high grade and a SiC particle is a raw material suitable for producing the specific non-oxide ceramics containing the ceramics like the Si₃N₄ association SiC and the Si association SiC. Moreover, when used as the silicon source with which the slurry produced from the diamond saw which consists of mixture of a very pure silicon chip and water is also mixed with SiC in production of the Si₃N₄ association SiC, it is the outstanding raw material to an Si₃N₄ ceramic ingredient. These ceramic ingredients (as side-attachment-wall refractories in an aluminum fire-refining place), It is known that it is suitable for the application in the elevated temperature under the conditions experienced in metallurgical industry. as monolithic tapping hole ***** in an iron alloy furnace -- etc. -- Moreover, it is suitable also as oven fixture ingredients (a setter plate, beam, etc.) in many other industrial applications for giving the resistance over corrosion to ceramic industry (baking of home earthenware, a sanitary ware, a tile, an electric insulator, etc.), and an OAT and the wear list in an elevated temperature.

[0015]

[Means for Solving the Problem] Two main steps in the usual process for manufacturing such a ceramic ingredient are the powder tamping step generally called shaping and an elevated-temperature step generally called sintering or baking. Shaping means applying and forming a pressure or putting into a mold and building. The particle of a raw material is hardened at this step by the configuration which resembled the form of an end product somewhat. As an example of the general technique applied, powder compression moulding technique (1 shaft application of pressure or isostatic pressing), a slip casting method, an extrusion-molding method, and the injection-molding method can be mentioned. At a sintering step, a ceramic ingredient appears as the result of a chemical physics process (pure sintering), or a result of the combination (reaction sintering) of a chemical physics process and a chemical reaction.

Infiltrating in a liquid ingredient occurs as another possibility. A ceramic raw material must easy-ize a tamping step and a shaping step, and as far as the property and workability of an ingredient are concerned, it must bring about the high product of integrity.

[0016] It is predicted that it can embellish as a ceramic raw material for acquiring a special material property in a final ceramic product as a ceramic raw material for easy-izing the specific fabricating method for Si-SiC powder mixture. Probably, this includes techniques, such as other methods of corning for easy-izing spray drying, the specific half-desiccation fabricating method or the specific wet compaction method, etc. for easy-izing desiccation application of pressure. Moreover, adjusting the ratio so that one side of Si or SiC(s) which are a component from the first may be added so much more and it may be suitable for a special application is also predicted. Furthermore, adding little chemical elements, such as a dopant, as a means for acquiring the special property of a final product or adding a particle, a crystal whisker, or other ceramic raw materials with the little gestalt of a fiber, in order to produce ceramic complex are also predicted.

[0017] In order to apply slurry trash as a raw material for producing the ceramics, the solid-state fraction which consists of an Si particle and a SiC particle must be separated from the glycol or oil which is a liquid fraction. In both cases, the usual processes, such as sedimentation, a decantation, and filtration, can perform this. After separation, the liquid fraction which consists of oil or a glycol finds out a new application in a process-oriented industry, or it can return to photovoltaics industry and/or semiconductor industry, and it can recycle it.

[0018] However, the liquid residue exists, and unless it is after removing this liquid residue, a solid-state fraction cannot usually be used for the solid-state fraction after the first separation as a ceramic raw material. Typically, in both [of the slurry which used the glycol as the base, and the slurry which used oil as the base] case, the residue has a separation method in the range of 5 - 10wt% in sedimentation or the case by filtration.

[0019] In the case of the slurry which used the glycol as the base, final separation of a solid-state fraction and a liquid fraction can be easily carried out by heating precipitate and/or a filter cake even to the temperature of the range of 200-400 degrees C. The solid-state fraction obtained as a result consists of very pure mixture of Si particle and a SiC particle, and this mixture is suitable for carrying out reaction sintering to the gestalt of the Si₃N₄ association SiC enough. First, the mixture of a pure Si particle and a pure SiC particle is covered over application of pressure and a shaping step, a desired form succeeds in it, and after that, it is covered over the heating step under nitrogen-gas-atmosphere mind, and is heated by even the temperature within the limits of 1050-1450 degrees C. By Si particle reacting at such temperature according to following reaction-formula: $3\text{Si}(\text{solid-state}) + 2\text{N}_2(\text{gas}) \rightarrow \text{Si}_3\text{N}_4(\text{solid-state})$, the ceramic ingredient will be formed, when this Si₃N₄ joins together together with a SiC particle in a sintering process. In the case which uses the sludge produced from the diamond saw which includes a silicon chip and water, those silicon chips contain the thin layer of SiO₂, and in order make Si₃N₄ form and to expose Element Si to N₂, they must return this SiO₂. This can be attained by raising sintering temperature to 1400-1500 degrees C simply.

[0020] The typical presentations of the precipitate obtained as a result after separation of the beginning of the solid-state fraction of the slurry which used the glycol as the base, and/or a filter cake are an about 65 to 75 wt% SiC, a 15 - 25wt% Si, and a 5 - 10wt% glycol. On the other hand, in the case of the slurry which used oil as the base, those numeric values of the precipitate and/or a filter cake are an about 40 to 60 wt% SiC, a 30 - 50wt% Si, and 5 - 15wt% oil. Like point **, this is telling excelling in the ratio of an Si particle pair SiC particle forming the Si₃N₄ association SiC. The approach by this invention can be used to any mixture of Si particle and a SiC particle, and is range whose concentration of Si particle is 2 - 100wt% suitably, the range of it is 10 - 50wt% more suitably, and the range of it is 15 - 30wt% most suitably. It is desirable that the mixture accompanied by 15 - 30wt% Si particle uses such mixture in order to bring about the strongest and densest ceramic after sintering. It turns out that the presentation of the slurry produced from the typical presentation of the slurry obtained after clearance of the beginning of a liquid phase as most typical result after completion of clearance of a liquid phase has usually fallen within the range of this. This is one of the reasons which show that these slurries are suitable for

production of these ceramic ingredients enough.

[0021] In the case of the slurry which used oil as the base, even if the evaporating temperature of some fractions of the oil residues is too high and it heats the precipitate and/or filter cake, it may be thoroughly unremovable. Since installation of the oxygen into a ceramic ingredient is not desirable, heat-treatment must be carried out under an inert atmosphere. this is contained in the oil -- disassembly of that hydrocarbon or all hydrocarbons is brought about for how many minutes, and C element and H₂ are formed in a pyrolysis process. Moreover, as the result, the solid-state fraction which consists of an Si particle and a SiC particle is the range of 1 - 10wt% of the mixture typically, and will be mixed with C element. However, since C element is not desirable for many fireproof applications and/or metallurgical applications, it should eliminate existence of C element. But though C element is regrettable in order to form SiC [before reaching sintering temperature] with Si, this situation is altogether produced with nature in a sintering process. After all C is consumed, the only thing in which it should succeed certainly is that the first powder mixture contains sufficient Si particle as Si of sufficient amount to form ceramic association remains. When producing the Si₃N₄ association SiC from the slurry which used oil as the base, therefore, a typical process The preheat treatment under the nitrogen-gas-atmosphere mind in the temperature of 700-800-degree C order is included until a pyrolysis is completed. Subsequently In order to change all C elements into SiC, temperature is raised even to the range of 1050-1450 degrees C, Si which remained is changed into Si₃N₄ after that, and a ceramic is formed.

[0022] In addition to generation of the Si₃N₄ association SiC, the Si association SiC (SiSiC) can also be built from the slurry which used oil as the base. In this case, nitrogen-gas-atmosphere mind is transposed to an inert atmosphere and a type target by argon atmosphere. Moreover, in this case, first, by heating a slurry at about 700-800 degrees C, a hydrocarbon must be disassembled at a pyrolysis step, temperature is raised and a sintering process is started after that. The temperature requirement in a sintering process is intrinsically [as the case where the Si₃N₄ association SiC is formed from the slurry which used oil as the base] the same.

[0023] Sintering temperature must be adjusted according to the content of Si particle in mixture, and can become an elevated temperature 2000 degrees C or more. Such adjustment is known for this contractor. Like point **, it is desirable to apply a slurry so that Si particle and SiC particle mixture which are obtained as a result may include 5 - 25% of Si particle.

[0024] Furthermore, some shaping processes including an isostatic pressing method, a desiccation inflatable flexible bag technique, a slip casting method, a sintering process, an extrusion-molding method, etc. are also known as a process for fabricating these types of ceramics. The main concepts of this invention are utilizing slurry trash and producing these types of ceramic ingredient. Therefore, it should be thought that these addition products, the manufacture approach, and the ceramics obtained as those results are built into this invention.

[0025] thus, the thing substantially done for the reuse of all the liquid phases (a glycol or oil) in an industrial application -- moreover, the problem in connection with disposal is thoroughly solved by fully reusing the solid-state component (Si particle and SiC particle) of slurry trash, and forming a series of new ceramic products and non-oxide ceramics. However, it is also possible [it] further by forming in the shape of a briquette, and subsequently to a cupola iron manufacture furnace using those briquettes, putting them in as the source of silicon and carbon to reuse slurry trash directly by making it combine [in the case of the slurry which used oil as the base for the solid-state fraction of a slurry] with concrete association or water glass in the case of the slurry which used pitch association and a glycol as the base.

[0026]

[Embodiment of the Invention] Next, the example which shows a suitable operation gestalt explains this invention to a detail further. However, these examples never do not exist what was created as what restricts this invention.

[0027]

[Example 1] A;70wt% SiC which obtained the precipitate of the following presentations after separation of the beginning of the solid-state fraction of the slurry by the formation sedimentation of the Si₃N₄ association SiC from the slurry trash which used as the base the glycol produced from a wire saw, a

20wt% Si, and a 10wt(s)% glycol. Subsequently, the precipitate was pressurized in 1 shaft by the force of 100MPa(s), and the test sample whose dimension is 10x10x100mm was formed. It was airtight and those samples were paid to the vertical mold mullite lining tubular furnace of a water cooling type. Final clearance of a glycol carried out evacuation of the furnace, and was performed by heating those samples at 400 degrees C for 24 hours.

[0028] The furnace was filled with pure nitrogen by the pressure of Hazama from 1025mbars to 1050mbars among the nitridation, carrying out monitoring of the pressure. When nitrogen was consumed and the pressure descended even to the low-limit-setting point, nitrogen was supplied until it reached the high-limit-setting point. Heating of those samples was carried out according to the following time amount / thermometer drawings. Nitrogen was supplied after completion of clearance of a glycol, temperature was raised at a rate of 400 degrees C/hour until it amounted to 1100 degrees C, and it held at 1100 degrees C for 72 hours. Subsequently, further, it held at raising and 1150 degrees C for 24 hours, and temperature was held at 1200 degrees C for 6 hours for 48 hours at 1300 degrees C, respectively, and finally it raised to 1350 degrees C, and held for 48 hours.

[0029] Those test samples obtained as a result were characterized and carried out as the following after the nitridation.

- ISO Measurement of the bulk density by 5017, and the apparent porosity;
- ASTM Measurement [of cold-bend strength]; and -XRD powder diffraction by C 651-91.

[0030] The microphotograph of the ingredient is shown in drawing 1 . The field of bright gray is SiC, and the field of dark gray is Si₃N₄, and a black field is an opening.

[0031] The bulk density of those test samples is 2.53**0.00 g/cm³, the apparent porosity is 17.64**0.07%, and it became clear that bending strength was 57.4**6.36MPa. XRD powder diffraction analysis was carried out with the sample of the sample ground to <45micrometer particle size. Si₃N₄ is in the condition accompanied by 4 3Ns of beta-Si of a minute amount, and mainly consists of 4 3Ns of alpha-Si, and the analysis showed that SiC mainly consisted of alpha-SiC. Furthermore, it became clear that the ferrosilicon (Fe₃Si) of a minute amount also existed.

[0032] The consistency of the silicon nitride joint silicon carbide ingredient currently sold as goods is 2.6 - 2.7 g/cm³ typically, and the apparent porosity is 14.6 - 18%, and bending strength is about 60 MPa (s). Therefore, the ingredient by this invention has a little lower consistency, and porosity is in a certified value. However, bending strength is below an ingredient currently sold as goods. Those test samples showed the clear indication of interlaminar peeling which reduces bending strength considerably. Although the reason of interlaminar peeling now is not clear, it is thought that it is related to having pressurized the sample which has a probably comparatively high glycol content. Being able to strengthen [therefore] the ingredient easy more by adjusting application-of-pressure actuation, the slurry is fully suitable as a raw material for producing silicon nitride joint silicon carbide.

[0033]

[Example 2] A;50wt% SiC which obtained the precipitate of the following presentations after separation of the beginning of the solid-state fraction of the slurry by the formation sedimentation of the Si association SiC from the slurry trash which used as the base the oil produced from a wire saw, a 40wt% Si, and 10wt(s)% oil. Subsequently, the precipitate was pressurized in 1 shaft under the force of 160MPa (s), and the test sample of the shape of a cylinder whose dimension is 20x20mm was formed.

[0034] It was airtight, and those samples were divided into two batches and paid to the vertical mold mullite lining tubular furnace of a water cooling type. The one half of the beginning of those samples was heated at about 700 degrees C under argon atmosphere, and another one half was heated even at 1300 degrees C under argon atmosphere. Moreover, operation of a furnace was suspended, after heating the one half of the beginning of those samples to 700 degrees C with the heating rate of 150 degrees C/hour and holding to the temperature for 2 hours. On the other hand, after heating the second one half of those samples to 1300 degrees C with the heating rate of 150 degrees C/hour and holding to the temperature for 5 hours, the temperature was returned to lowering and a room temperature at a rate of 150 degrees C.

[0035] These test samples were also characterized by the following examinations, and were carried out.

- ISO Measurement of the bulk density by 5017, and the apparent porosity;
- ASTM Measurement [of low-temperature-grinding strength]; and -XRD powder diffraction by C 133-91.

[0036] The bulk density and the apparent porosity which receive both batches and which were obtained as a result are shown in a table 1. The table shows after sintering that the apparent porosity decreased 11%. It is not thought at all as an exception which is possible in the copper, the zinc, and the ferrosilicon compound of the minute amount produced from wear of the wire made from steel of a wire saw that a liquid phase may exist at such temperature.

[0037] The low-temperature-grinding strength of the sample belonging to the batch I whenever [stoving temperature / whose] is 700 degrees C was less than 3.5 MPas. As for the sample which belongs to the batch II whenever [stoving temperature / whose] is 1300 degrees C on the other hand, the low-temperature-grinding strength of 69.1**14.4MPa was shown.

[0038] The sample of the sample extracted from both batches is ground to <45micrometer particle size, and it is Phillips. PW 1710 The XRD powder diffractometer analyzed. The sludge is in the condition accompanied by that silicon and ferrosilicon a little, and the analysis result of the sludge (batch I) by which the pyrolysis was carried out showed that it mainly consisted of alpha-SiC. Moreover, the cristobalite of a minute amount also existed. On the other hand, a little, probably the sintered sludge (batch II) is in the condition accompanied by the quartz of *****, and had mainly become the cristobalite of that amount, a ferrosilicon, and a list from alpha-SiC.

[0039]

[Example 3] **** of the Si3N4 association SiC from the slurry trash which used as the base the water produced from a diamond saw -- this example is the same as an example 1 except for having to add SiC of the initial complement which the slurry used here contained only silicon and water, therefore was mentioned above in that slurry. Therefore, the liquid fraction which must be removed consists of water which can be easily removed with the same heating as the case of the slurry which used the glycol of an example 1 as the base. The only exception is having to return this SiO2 to Si element, in order for that moisture to bring SiO two-layer to Si particle and to form Si3N4. This reduction can be automatically attained by raising sintering temperature to 1400-1500 degrees C.

[0040]

[A table 1] Bulk density and apparent porosity of slurry precipitate which use the heat-treated oil as the

バッチ	加熱温度 (℃)	嵩密度 (g/cm ³)	見掛け気孔率 (%)
I	700	1.89 ±0.01	40.28 ±0.26
base II	1300	2.04 ±0.01	29.48 ±0.65

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CLAIMS

[Claim(s)]

[Claim 1] The slurry trash which includes the liquid with which it is an approach for manufacturing non-oxide ceramics, and : and the aforementioned approach consist of the mixture, the oil, or the glycol of Si particle and a SiC particle is used.;

- In order to remove all of the aforementioned slurry trash, or almost all the liquid fraction, form said non-oxide ceramic ingredient, covering the powder mixture which covered said slurry trash over the separation process, and was obtained as; and a - result over the usual process.;

The manufacture approach of non-oxide ceramics characterized by things.

[Claim 2] The approach according to claim 1 that most liquid fractions of said slurry trash are characterized by dissociating from a solid-state fraction by sedimentation and the subsequent decantation.

[Claim 3] Step from which the manufacture process removes the glycol residue in precipitate with moderate heating under : and a vacuum when said approach is applied, in order to manufacture nitride joint silicon carbide from the slurry trash which used the glycol as the base;

- Step which sinter the ceramic ingredient by heat at a certain temperature under nitrogen gas atmosphere mind that the silicon nitride which said Si particle react the body which succeed in step; and the - aforementioned configuration which fabricate the mixture of Si particle obtained as a result and a SiC particle on the body which succeed in a certain configuration with N₂, and combine said SiC particle together be form;

The approach according to claim 2 characterized by including.

[Claim 4] Said moderate heating for evaporating : and the aforementioned glycol residue in said approach is carried out at the temperature of 200-400-degree C order.;

- The aforementioned tamping is carried out by the force of the order of 100MPa, and; and the - aforementioned sintering are carried out in the temperature of the range of 1050-1350 degrees C by the nitrogen pressure of 105Pa order.;

The approach according to claim 3 characterized by things.

[Claim 5] When said slurry contains Si particle and water which are produced from a diamond saw, the great portion of : and aforementioned water are removed by sedimentation and the subsequent decantation.;

- The remaining water is removed by heat treatment in 200-400-degree C order.;

2 - 100wt% Si particle and since more suitably obtains 10 - 50wt% Si particle and the mixture which contains 15 - 30% of Si particle most suitably, suitably adds a SiC particle to said slurry.;

- The aforementioned tamping is carried out by the force of the order of 100MPa, and; and the - aforementioned sintering are carried out in the temperature of the range of 1400-1500 degrees C by the nitrogen pressure of 105Pa order.;

The approach according to claim 1 characterized by things.

[Claim 6] separation of the greater part of : when said slurry trash uses oil as the base and the mixture of Si particle and a SiC particle, and mineral oil is included, and its liquid phase -- sedimentation and the

decantation after it -- including --;

- heating said precipitate under an inert atmosphere until the hydrocarbon in said oil decomposes by the pyrolysis and removal of the oil residue forms a carbon element -- including --;
- Form the ceramic ingredient by sintering at a certain temperature under nitrogen-gas-atmosphere mind which forms the silicon nitride to which Si particle which C element reacted with Si the body which fabricates the powder mixture obtained as a result on the body which succeeds in a certain configuration, and succeeds in; and the - aforementioned configuration, and formed SiC, then remained reacts to with N₂, and sinters a SiC particle together.;

An approach given in claims 4 and 5 characterized by things.

[Claim 7] In said approach, it is the range whose : and aforementioned pyrolysis temperature are 700-800 degrees C, and is held for 2 hours at the temperature of this range.;

- The aforementioned tamping is performed by the force of the order of 100MPa, and; and the - aforementioned sintering are carried out in the temperature of 1050-1350-degree C order by the nitrogen pressure of 105Pa order.;

The approach according to claim 6 characterized by things.

[Claim 8] Step in which the manufacture process removes the oil residue in the precipitate by : and the pyrolysis, and forms C element when being applied, in order that said approach may manufacture silicon nitride joint silicon carbide;

- Said Si particle reacts the body fastened and hardened with introduction C, and form SiC. step; which fabricates the mixture of Si particle and the SiC particle which were obtained as a result, and C element on the body which succeeds in a certain configuration, and - subsequently

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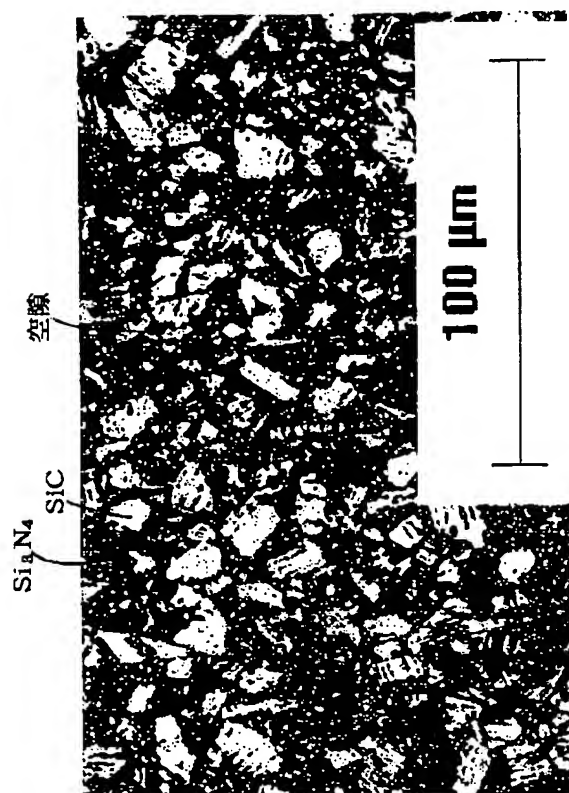
DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] Drawing 1 shows the microphotograph of the Si₃N₄ association SiC produced by the approach by this invention. The field of bright gray is SiC, and the field of dark gray is Si₃N₄, and a black field is an opening.

[Translation done.]

Drawing selection [Representative drawing ▾]



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